

Effects of expansive additive on cement composite properties

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Abstract. Shrinkage is a due to chemical and physical processes in concrete. The results of these are volume changes that may cause cracking, deformation, decrease in strength and especially durability of a construction. The amount of water in concrete, the fine particles content, the dimension of construction, the method of curing of the fresh concrete and environmental conditions (temperature, humidity) are some factors influencing the shrinkage of concrete. This article deals with reduction the shrinkage of cement composites by addition of expansive cement Denka CSA and synthetic gypsum. The additives were used at a dosage of 10% of the original weight of cement. The obtained results showed different impact of these additives to the volume changes and the compressive strength of cement composites. The Denka CSA additive had a positive effect to monitored properties of cement mortars. It caused the lowest shrinkage; slight increasing of long-term compressive strength and decreasing of bulk densities after 2, 28 and 90 days of hardening.

1. Introduction

The first mention of composite materials dates back to the ancient world. The composite material is a material consisting of two or more components with different properties. These components give the resulting material novel properties, which is not in itself any of its components. These materials mainly consist of a filler and binder. The binders may be different, but most often the cement composites are based on Portland cement clinker. Nowadays, when modern materials are produced, concrete due to its mechanical, physical, chemical and structural properties belongs to the most used building materials. In the design and realization of concrete elements and structures it needs to be focused on the volume changes occurring in the concrete. They have a significant effect on later properties. These changes are: shrinkage, swelling, creeping and volume changes from the temperature effect. Inherent characteristic of any concrete and other cement-based composites is its shrinkage.

Shrinkage of cement composites can be characterized as a consequence of reducing the moisture content of the porous structure. Dependence mainly affects the capillary forces exerted in the composite. These forces compress the cement composite from all sides, resulting in its shrinkage [1].

Shrinkage generally depends on: the dosage of water in the concrete, the fine particle content, dimension of construction, the method of fresh concrete curing, the age of the concrete and the environmental conditions (temperature, relative humidity, pressure and air velocity) [2]. The shrinkage of concrete can be divided into 2 main stages: the early and the long term ages. The early stage is commonly defined as the first day (within the first 24 hours), while the concrete is setting and starting to harden. After hardening of concrete, the water which has not been consumed by cement hydration leaves the pore system, in case there is not a balance between the moisture content in the composite and the surrounding environment. While drying, capillary forces are formed in the pore system together with the surface tension of water causing reduction of the pore and material volume. This is known as drying shrinkage [2-3]. The next more relevant type of shrinkage is autogenous shrinkage.



There are many ways of reducing the shrinkage of cement composites. The one of the ways is suitable reinforcement of concrete. Different types of dispersed fibers can be used to eliminating the effects of shrinkage of self-leveling cement screeds [3-5]. The next option is to use such an additive inorganic material as the fly ash and the shrinkage reducing agent (SRA) [6-7]. This SRA is a kind of chemical substance that helps reducing the surface tension by dropping the capillary stress and, hence, reducing the shrinkage of the paste in terms of both the chemical reaction and the external environment. The expansive additives generally help reducing the shrinkage due to many different factors, for example, the enlargement from water absorption and the forming of the pore and crystal structure in the process of hydration reaction [8-9].

Calcium sulfoaluminate cement (CSA cement for short) was developed in Japan. This special cement distinguishes itself from Portland cement by a high-speed bonding, fast strength development, and a shrinkage reduction. CSA cement has been used for decades as a binder in concrete for bridges, airport start- and runways, concrete road-repair, and many other applications where a quick reuse is required. Nowadays, CSA cement is also used in dry mortars for self-levelling floors, levelling compounds, casting mortars, tile adhesives, grouts, etc. [10-11]. It is produced by crushing a sintered compound mainly consisting of limestone, gypsum, and bauxite (calcium oxide, calcium sulfate and aluminum oxide) in adequate amounts. The chemical composition of sintered CSA clinker mainly consists of haulyne, free lime and free gypsum. Hydration of these minerals forms ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$), a very small crystal with dimensions of several microns (figure 1). In the cement paste hardening process, this colloid-like crystal becomes burr-like within minute gel voids. It thus reduces hardening shrinkage and dry shrinkage of the gel, and furthermore it acts to expand the gel. Therefore, the presence of ettringite increases the density of the concrete matrix, reduces dry shrinkage, and induces compressive stress into a concrete structure under a restrained condition. Through the above mechanism, CSA reduces dry shrinkage cracking in concrete and increases the water-tightness of concrete structures [12].

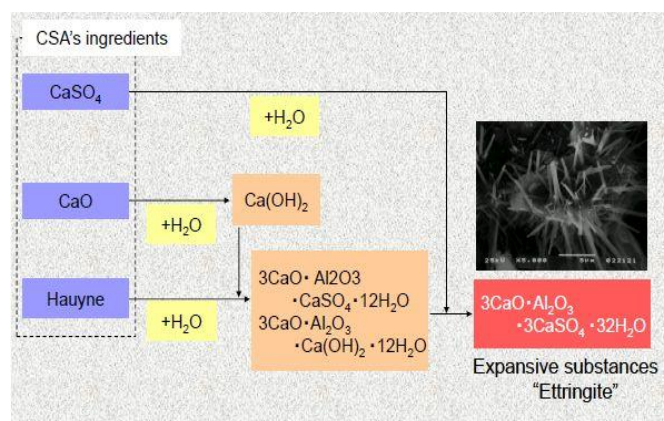


Figure 1. The hydration system of DENKA CSA [12].

2. Materials and mixture design

The experimental study was focused on the reduction of shrinkage of cement mortars by using the expansive cement Denka CSA and synthetic gypsum.

Cement CEM II A/S 42.5 R, in according with STN EN 197-1 [13], with following properties: compressive strength after 2 days 21.6 MPa, after 28 days 49.7 MPa, initial setting time 200 min, setting time 250 min and soundness (expansion) by Le Chatelier 7.2 mm.

Natural quarried aggregate divided by sieve analysis on fraction 0/1, 1/2 and 2/4 mm.

Fly ash is characterized as the gray powder with apparent density from 700 to 900 $\text{kg} \cdot \text{m}^{-3}$; activity index after 28 days min 75%; activity index after 90 days min 85% and other characteristics declared

by producer. The chemical compositions of fly ash (wt. %) are: SiO₂ (38.76); Al₂O₃ (16.08); Fe₂O₃ (3.21); CaO (21.28); MgO (1.98); TiO₂ (0.51); SO₃ (13.99).

Water supplied by the public water supply company was used in the mixtures. All cement mortars had w/c ratio = 0.55.

The agent *MS 4-12*, based on modified polycarboxylates, was used for reference mortar (labeled REF). It is characterized by density at 20 °C 1060 kg.m⁻³ ± 20 kg.m⁻³; pH value at 20°C 6,0 ± 2.0; dry matter content 20% ± 1%; chloride content max 0.10% by weight of agent. Furthermore, *BR stabilizer 101* with density 1026 kg.m⁻³, 3.6% dry weight and pH 9.5 was used.

Expansive cement *DENKA CSA* is Calcium sulfoaluminate cement (CSA) cement produced from a special clinker and milled to the particle size of cement. It is off-white powder with specific density ca. 2860 kg.m⁻³; loose apparent density from 800 to 900 kg.m⁻³; tapped apparent density ca. 1500 kg.m⁻³ and specific surface by Blaine ca. 370 m².g⁻¹. Synthetic gypsum or calcium sulfate (gypsum) is the end product of flue gas desulphurization. It represents chemically very pure material with a purity of 97-98% which is mainly used in the manufacture of cement and plaster. Further application is in the manufacture of plaster mixtures, aerated concrete and as a setting regulator. It is characterized by the moisture content of 10%; pH value of 6 – 9 and purity of 95%.



Figure 2. The expansive cement Denka CSA.



Figure 3. The synthetic gypsum.

2.1. Mixture design

The design of cement mortar composition was based on commercially produced self-leveling cement screeds. Reference mortar was modified by expansive additive at a dosage 10% of weight of cement. The proportions of cement mortars are given in table 1.

Table 1. The overview of cement mortars mixing ratios to cubic meter.

	Reference mortar (kg.m ⁻³)	Modified mortar (kg.m ⁻³)
Cement	243.15	218.84
Expansive additive		
- Denka CSA	-	24.32
- Synthetic gypsum	-	24.32
Fly ash	154.00	154.00
Aggregate		
- fraction 0/1	518.72	518.72
- fraction 1/2	207.49	207.49
- fraction 2/4	311.23	311.23
Plasticizing agent		
- MS 4-12	6.00	6.00
- BR stabilizer 101	6.24	6.24
Water	218.84	218.84

3. Measuring methods

Cement mortars were mixed in a normalized laboratory mixer. After that, the properties of the fresh mortars were determined: consistency of the mortar (determined by a flow test on the Haegemann flow table) and the bulk density. The test samples to determine compressive strength were cylindrical with diameter and heights of 30 mm and were cured in humid environment. The compressive strength of hardened mortars was measured after 2, 28 and 90 days. The test samples to determine shrinkage were beams of 40 x 40 x 160 mm with glass contacts in front. They were cured for 24 hours in humid environment. After this time, samples were unmolded and the initial length measurements were done. Subsequently, the samples were kept in a laboratory environment (temperature $20^{\circ}\text{C} \pm 5^{\circ}\text{C}$, relative air humidity $\phi = \text{ca } 50\%$). The drying shrinkage of cement mortars was determined as loss of length compared to the initial length determined after 24 hours of curing in moist environment. Shrinkage was measured after 2, 4, 7, 14, 28, 56 and 90 days of curing in the laboratory environment. The length measurement was done using the Graf-Kaufmann device. The weight loss was measured on the same samples used for the shrinkage measurements.

4. Results and discussion

4.1. Consistency and bulk density

The measured values of the consistency and the bulk density of fresh cement mortars for each mixture are given in table 2.

Modifications of cement mortars with expansion additives caused a slight worsened consistency compared to the reference sample (REF). Flow diameter ranged from 233.5 to 244 mm. The expansive additives had a different impact on the bulk density of fresh mortars. Replacing the cement with expansive cement Denka CSA (labeled PC) led to a slight increase in bulk density to value 2140 kg.m^{-3} compared to the reference mortar. Using the synthetic gypsum (sample labeled PE) had an opposite effect; reducing bulk density to value 2000 kg.m^{-3} .

Table 2. Properties of fresh cement mortars.

	Bulk density (kg.m^{-3})	Consistency (mm)
REF	2120	282.5
PC	2140	244.0
PE	2000	233.5

4.2. Shrinkage and weight loss

Measured values of shrinkage, respectively swelling of hardened cement mortars are given in figure 4. Using the expansive additives had a various impact to the volume changes of the hardened cement mortars. Shrinkage of the reference cement mortar and mortar with synthetic gypsum additive was approximately the same during 14 days. The Denka CSA additive caused swelling of the mortars in the first days. In other monitoring days, the gradual shrinkage of samples was already observed. The total value of shrinkage of the reference mortar after 90 days was ca. 0.74‰; mortars modified by additive Denka CSA ca. 0.68‰ and synthetic gypsum ca. 0.79‰.

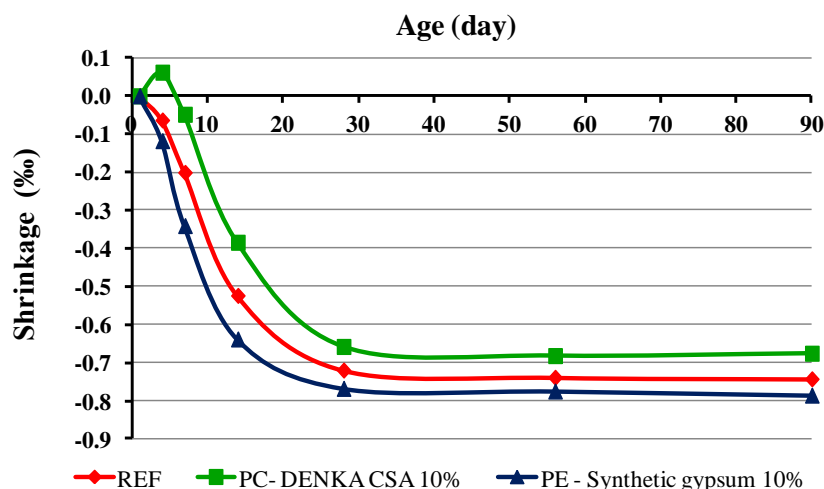


Figure 4. Shrinkage of the reference sample and samples with 10% doses of expansive additive.

4.3. Compressive strength

Application of expansive additives had impact on the physical properties of hardened cement mortars, which can be seen in table 3.

Table 3. The results of hardened cement mortars.

	Bulk density (kg.m^{-3})			Compressive strength (MPa)		
	2 days	28 days	90 days	2 days	28 days	90 days
REF	2160	2010	1990	9.85	24.38	26.00
PC	2050	1910	1870	11.02	23.09	27.92
PE	2030	1710	1690	6.11	15.94	20.06

Using additive Denka CSA resulted in only a slight increase in the initial (2-day) and the long term (28 and 90 days) compressive strength of cement mortars compared to the reference mortar. The values of compressive strength in the monitoring period ranged from 11.02 MPa to 27.92 MPa. The values of bulk density of cement mortars were less than the reference mortar after 2, 28 and 90 days of hardening.

Cement mortar modified by synthetic gypsum additive showed significantly the lower strength characteristics compared to the reference and the second modified mortar. The compressive strength values ranged from 6.11 MPa to 20.06 MPa in the same monitoring period. The bulk density values achieved the lowest values compared to other studied cement mortars.

5. Conclusion

Effect of using expansive additives to cement mortars were studied and recorded in this research. The achieved results are summarized hereinafter:

- Application of the expansive cement Denka CSA led to reduce the shrinkage of cement mortars and the complete elimination of shrinkage in the first days of hardening. The significant benefit is also the increase of the initial and the long term compressive strength. The values of compressive strength ranged from 11.02 MPa to 27.92 MPa after 2, 28 and 90 days of hardening. The dosage of CSA cement did not have significant impact on the bulk density.

These values are very similar in comparison to values of samples modified by synthetic gypsum additive.

- Synthetic gypsum additive had negative effect on the properties of cement mortars. It caused increasing the shrinkage (ca. 0.74% after 90 days of hardening) and also a significant the compressive strength reduction (from 6.11 MPa to 20.06 MPa after 2, 28 and 90 days of hardening). The positive effect of this additive was reflected in the decreasing values of bulk densities compared to other studied cement mortars. The reference mortar's bulk density values after 2, 28 and 90 days of hardening ranged from 2160 kg.m⁻³ to 1990 kg.m⁻³; the cement mortar values modified by Denka CSA cement ranged from 2050 kg.m⁻³ to 1870 kg.m⁻³ and cement mortars with synthetics gypsum additive ranged from 2030 kg.m⁻³ to 1690 kg.m⁻³.

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